Hierarchically Organized Nanocomposites for Enhanced Fatigue Life of Rotorcraft Components

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ABSTRACT

Rotorcraft components, which are often made with reinforced fiber composites, are subjected to severe fatigue loadings due to increased performance demands. Therefore, considerable research interest exists in improving fatigue life of conventional fiber reinforced composites. Nanocomposites are a new class of materials which seek to improve mechanical performance of materials by creating nanoscale crack-nanofiller interactions. In this study we demonstrate the fatigue life improvement of conventional composites by addition of SiO_2 nanofillers. The epoxy resin was initially modified with nanofillers to test the static fracture toughness. Once the improvement in static facture toughness was confirmed, three phase modified fiber reinforced composites were made using the modified resin. Cyclic tests were performed at various stress level which demonstrate that three phase nanocomposites perform better than conventional fiber reinforced composites. Fractographic analysis suggests that nanofiller de-bonding from the matrix as well as crack deflection around nanofiller clusters contributes to the improved fracture toughness and fatigue life.

INTRODUCTION

Rotorcrafts are entering their next generation with increased demands on their load carrying capacity as well as frequency of operations. Consequently, their safety and reliability must be evaluated against new challenges. Severe cyclic loads in the form of vibrations pose major design constraints on the rotorcraft components and fatigue is one of the principal causes of mechanical failure. Hence there is considerable interest in exploring new classes of materials with enhanced fatigue life.

Carbon fiber reinforced polymers (CFRP) have been conventionally used in rotorcrafts for their high strength to weight ratio. Carbon fibers carry the bulk of the load, while the weaker polymeric matrix material holds them together. It is observed that the fatigue failure of CFRP originates in the matrix at the location of microscopic flaws such as voids and microcracks. The next phase of failure is characterized by stable growth of these sub-critical microflaws. This phase is followed by the rapid catastrophic failure in form of unstable crack growth across matrix fiber interphase (Ref. 1).

Nanocomposites are a relatively new class of materials which show potential in enhancing mechanical properties (Ref. 2). Nanocomposites are typically manufactured by the addition of nanoscale fillers like SiO_2 (Ref. 3), carbon nanotubes (Ref. 4), graphene oxide (Ref. 5) etc. to the resins used as matrix materials. These nanofillers create interaction with the subcritical cracks in resin matrix which would consequently improve the fracture toughness. The modified resin can be used instead of regular resin to create three phase nanocomposites (three phases being matrix, fiber and nanofillers).

Researchers have demonstrated fracture toughness enhancement of thermoset polymers modified by the addition of nanofillers (Ref. 6, 7). However, fatigue studies of three phase composites manufactured using modified polymers are rare. In the following sections we describe the manufacturing and testing of such nanocomposites and discuss the results.

MATERIAL AND EXPERIMENT DETAILS

Nanocomposite preparation

Epoxy (Diglycidyl ether of Bisphenol A) is a thermoset polymer resin which is commonly used as matrix in CFRP. Commercially available epoxy resin and hardener system provided by Fiber Glast (resin 2000-B and hardener 2120-B)

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was used in the study. Resin was modified by addition of SiO_2 (silica) nanoparticles. The silica nanoparticles were obtained from Sigma Aldrich (Fumed, Silica S5130). Individual particles are sized at ~7 nm which form aggregate chains of 10 to 30 spheres in length.

High viscosity of the resin and the very high specific surface area of the nanoparticles make uniform dispersion of nanoparticles difficult. Hence, nanoparticles were first dissolved in a low viscosity solvent and sonicated for 90 minutes to obtain a uniform dispersion. The resin was added to the mixture and sonicated for additional 90 minutes. The solvent-rich mixture was heated to evaporate the solvent and further kept in vacuum at 70°C to remove any traces of the solvent. The modified resin thus obtained was mixed in predetermined quantity of hardener which initiates curing process (Figure 1).

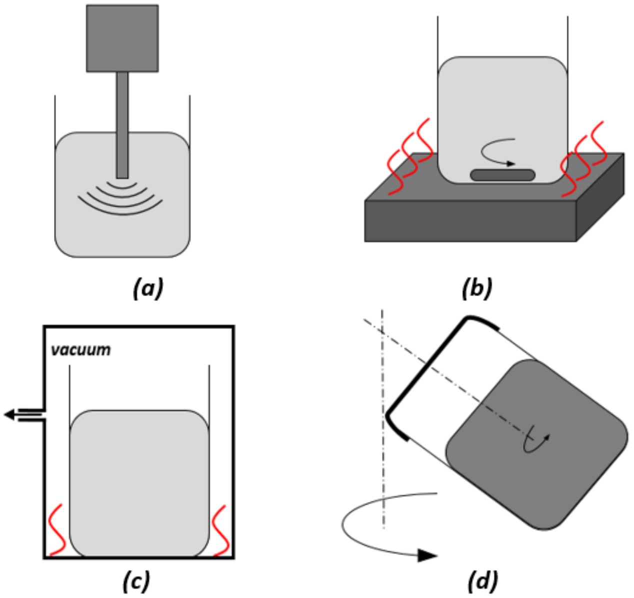


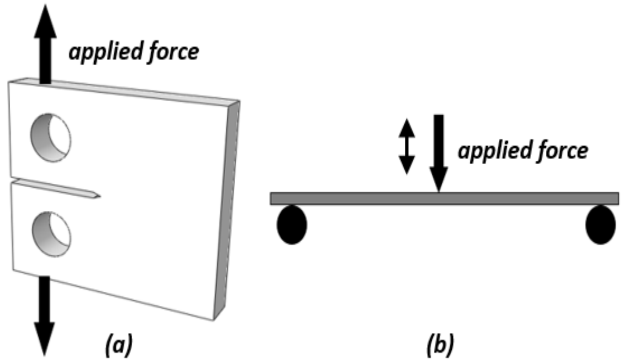
Figure 1. Preparation of modified resin: (a) sonication to achieve desired dispersion, (b) heating to evaporate solvent, (c) vacuum heating to get rid of solvent traces, (d) Mixing resin and hardener using planetary mixer

For fracture toughness tests, the resin and hardener mixture was poured in molds to create compact tension (CT) specimen. For fatigue tests, specimen were prepared using wet lay-up approach. Multiple carbon fiber sheets were uniformly wetted by modified resin-hardener system and stacked at 0^o-90^o orientation to get the desired specimen thickness. The layup was vacuum pressed and cured to obtain three phase composite plates.

Experimental details

To measure the fracture performance of the modified resin three different nanoparticle weight fractions of 5%, 7.5% and 10% were tested. Unmodified resin specimen were tested as well to establish a baseline. Three samples were tested for each loading fraction. A micro-crack was introduced by tapping a sharp razor blade at the root of CT specimen notch. The notched specimen was loaded uniaxially to failure, in accordance with ASTM 5045 (Figure 2a) (Ref. 7).

After establishing the fracture performance, fatigue tests were performed in accordance with ASTM D790 (Ref. 8). Two separate batches of CFRP made using unmodified resin (baseline) and modified resin with 5% weight fraction were tested to compare fatigue performance. Simply supported specimen of dimension10×100×2.5 mm^3 were centrally loaded in a cyclic fashion (Figure 2b). The tests were performed in force control mode with load ratio ($\sigma_{min}/\sigma_{max}$) of 0.1 and frequency of 3 Hz. Three different loading levels of $\sigma_{max}/\sigma_{failure}$ of 0.8, 0.75 and 0.7, where $\sigma_{failure}$ is the bending stress at failure in a static flexural test. Three samples were tested at each stress level.



. **Figure 2. Tested specimen: (a) CT specimen for fracture toughness test, (b) experimental setup for flexural cyclic loading test**

RESULTS AND DISCUSSION

Static fracture toughness

The static fracture toughness tests showed that addition of silica improved the fracture toughness of epoxy. The Mode I fracture toughness (K_{Ic}) of baseline epoxy was measured at 1.65 $MPa\sqrt{m}$. There was improvement relative to this baseline of 12%, 27% and 40% for loading fractions of 5%, 7.5% and 10% respectively (Figure 3). Although we observed continuous improvement with the loading fraction, the viscosity of the resin was observed to increase considerably for filling beyond 7.5% loading. This creates considerable difficulties in producing CFRP through the wet layup procedure.

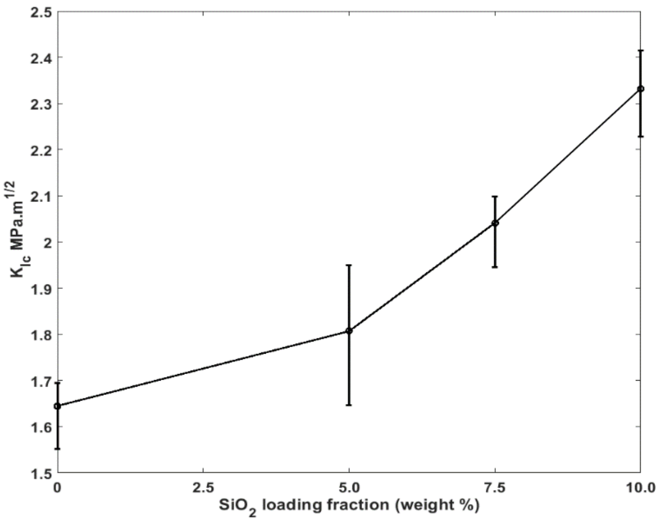


Figure 3. Fracture toughness of modified epoxy resin at different loading fractions.

Fractographic analysis (performed after static tests)

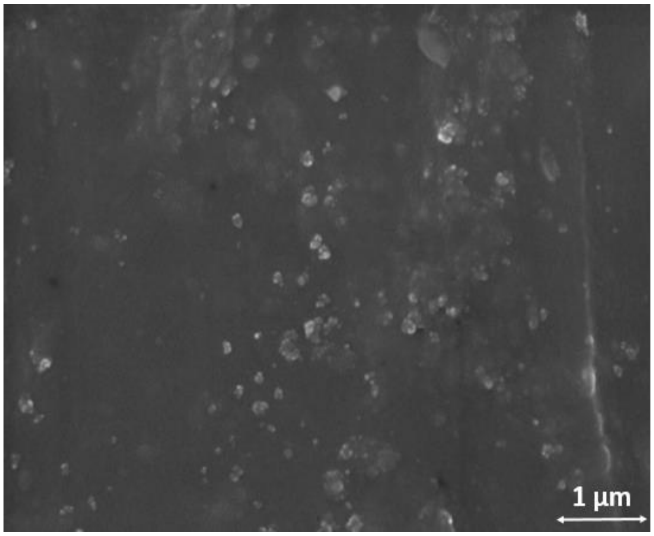


Figure 4. Distribution of SiO_2 nanofillers in epoxy

Uniform distribution of nanofillers is critical for the mechanical performance of nanocomposites as nanofillers might otherwise agglomerate to form stress concentrating flaws. The microscopic analysis of the fracture surface revealed that the nanoparticles were dispersed in clusters of ~120 nm diameter throughout the matrix (Figure 4).

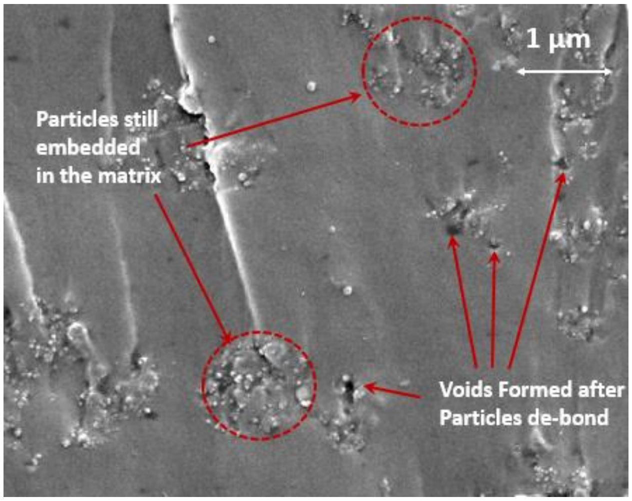


Figure 5. Several sites showing nanoparticle clusters and voids formed within clusters after the de-bonding of SiO_2 nanofillers during fracture

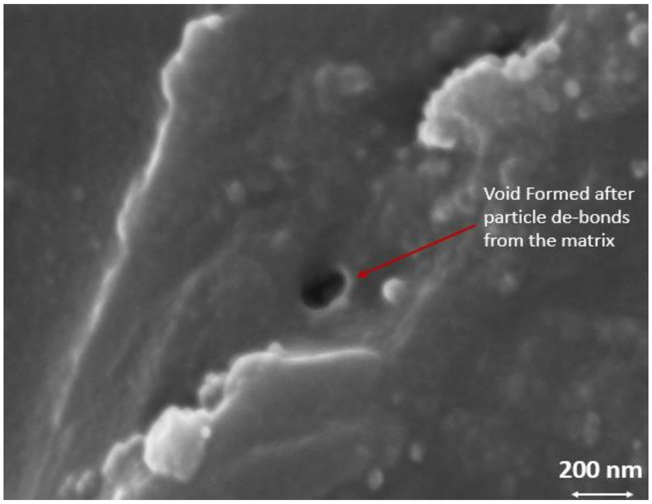


Figure 6. Nanoscale voids formed after the de-bonding of SiO_2 nanofillers during fracture

The scanning electron microscopy (SEM) performed at nanoscale exhibited several sites with voids of the size of nanofiller clusters (Figure 5, 6).

This indicates, as the crack propagates utilizing the energy provided by the external load, a part of that energy is spent in de-bonding the nanofillers from the matrix. This imparts toughening to the matrix. Additionally, the nanoscale voids created after the de-bonding also create pockets of nanoscale plasticity which adds to the toughening improvement.

Cyclic flexural tests

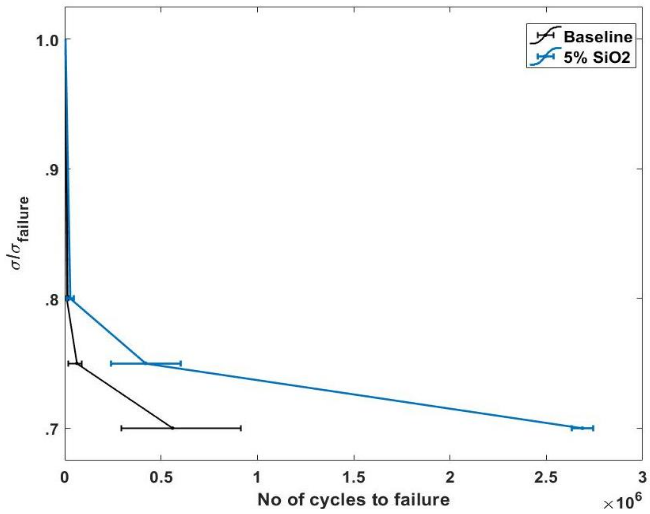


Figure 7. Comparison of S-N curves for baseline CFRP and CFRP with modified resin, the vertical axis has been normalized by $\sigma_{failure}$

Three phase composites with 5% SiO_2 were prepared to study the fatigue life. Baseline for fatigue tests were conventional CFRP prepared using unmodified epoxy. Figure 7 shows the S-N curve comparison for the baseline CFRP and modified three phase composite. Improvement in the fatigue life was observed at different stress levels. Furthermore, the improvement in fatigue life was increasingly more pronounced at lower stress levels, under high cycle fatigue conditions. This is critical from the perspective that rotorcrafts components are typically subjected to low stress levels for extended periods of time during regular operation.

Fractographic analysis of fatigue tests

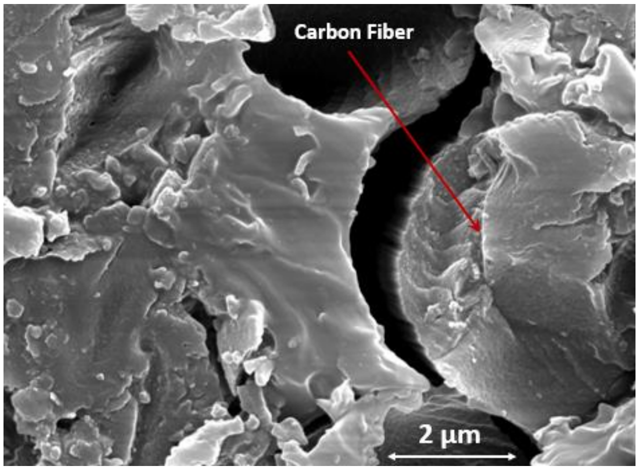


Figure 8. Fiber matrix de-bonding in three phase composite

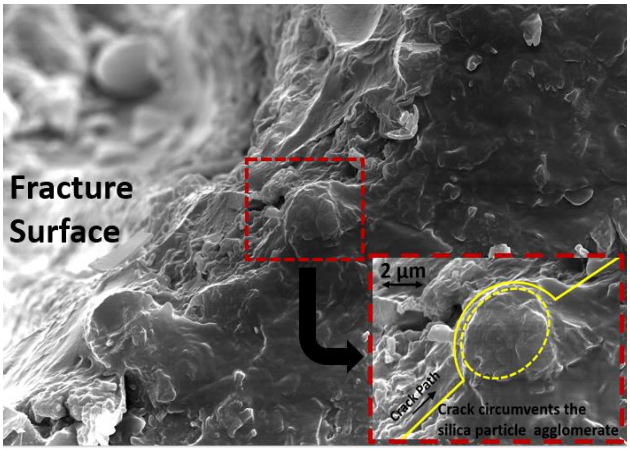


Figure 9. Crack defection around nanofiller clusters

SEM imaging of the fracture surface of the composites show features expected in fiber matrix composites like fiber matrix de-bonding (Figure 8). However, these mechanism are present in both baseline and modified resin composite hence they do not explain the improved fatigue performance of three phase composites.

The fracture surface of the three phase composites exhibits crack deflection around cluster of nanofillers (see Figure 9 for a typical image). The deflection of crack around clusters imparts toughness and slows down or retards the crack propagation. This crack deflection phenomena in conjunction with the previously mentioned nanofiller de-bonding would delay crack initiation in the matrix and retard crack propagation along the carbon-fiber/matrix interphase.

FUTURE WORK

We have demonstrated fatigue life improvement by modifying the epoxy resin with 5% loading of SiO_2 nanofillers. Different loading fractions of SiO_2 will be used in the future to create new batches of three phase composites. Comparison of fatigue life performance of different loading fractions would help in determining optimum loading fraction for the system.

Graphene has attracted considerable attention recently in the nanocomposite's community (Ref. 9). Individual graphene platelets can have surface area of the order of microns, while being only few atomic layers thick (Ref. 10). The micron scale lateral dimensions of the graphene could potentially help in retardation of sub-critical crack growth, improving the fatigue life considerably. Graphene has been shown to improve static fracture toughness of thermoset polymers. Furthermore, the toughness improvement is achieved at very low loading fractions of less than 0.1% (Ref. 11). This provides added benefit of weight reduction in the final non-composite components. Although there would be a cost penalty since commercially available graphene is orders of magnitude more expensive compared to SiO_2. Future work will involve testing three-phase composites with graphene to compare the fatigue performance of the nanocomposite to that of SiO_2 nanofillers.

The carbon-fiber/matrix interphase is critical to the performance of CFRP. As the micrographic analysis has shown, the catastrophic failure occurs through crack propagation between the carbon-fiber and matrix. Therefore, changing the properties of the carbon-fiber/matrix interphase could drastically improve the fracture performance of the nanocomposite. Depositing nanofillers directly on the carbon fibers could potentially enhance the interface strength by providing additional surface area for higher effective load transfer between the matrix and the carbon-fibers. Obtaining uniform dispersion of nanoparticles in high viscosity reins is one of the vexing issues in nanocomposite manufacturing. In fact, the variability of their performance in different studies can be largely attributed to different qualities of nanofiller dispersion (Ref. 12). If nanofillers could be successfully deposited on fibers it would also simplify the nanocomposite manufacturing greatly, since achieving uniform dispersion in the matrix would no longer be necessary. We would be exploring different ways to deposit nanofillers directly on the carbon-fibers in future as it provides a promising way to improve conventional CFRP (Ref. 13, 14) in a simplified and consistent manner using nanofillers.

CONCLUSIONS

In this study we have demonstrated that-

1. Addition of SiO_2 nanofillers to thermoset polymer improves its fracture toughness
2. The modified thermoset can be used to create three phase composites with enhanced fatigue life
3. The enhancement of fatigue life is increasingly more pronounced at low stress levels under high cycle fatigue conditions.
4. Fractography of the fracture surfaces suggests that de-bonding of nanofillers as well as crack deflection are responsible for the improvement in toughness and fatigue life of to the composite

We will be performing fatigue tests with different SiO_2 loading fractions and using graphene in the future.

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